

THAT WHICH IS CLAIMED:

1. A method for the in-situ removal of impurities from a saponified solution of pyridine-2,3-dicarboxylic acid ester, said method comprising the steps of:

5 providing a saponified solution comprising a pyridine-2,3-dicarboxylic acid ester and a base;
reacting said solution with an oxidizing agent in an amount effective to remove impurities, thereby providing a purified saponified solution; and
collecting said purified saponified solution.

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2. The method of Claim 1 wherein said base is a hydroxide.

3. The method of Claim 2 wherein said hydroxide is sodium hydroxide.

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4. The method of Claim 1 wherein said oxidizing agent is selected from the group consisting of peroxides, peroxyacids, and hypohalite salts.

5. The method of Claim 4 wherein said oxidizing agent is hydrogen peroxide.

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6. The method of Claim 4 wherein said oxidizing agent is sodium hypochlorite or sodium hypobromite.

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7. The method of Claim 5 wherein said amount of hydrogen peroxide effective to remove impurities is an amount in the range of about 0.1 to about 2.0 moles hydrogen peroxide per mole of pyridine-2,3-dicarboxylic acid ester.

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8. The method of Claim 5 wherein said amount of hydrogen peroxide effective to remove impurities is an amount in the range of about 0.2 to about 0.8 moles hydrogen peroxide per mole of pyridine-2,3-dicarboxylic acid ester.

9. The method of Claim 1 wherein said amount of oxidizing agent effective to remove impurities is an amount necessary to change the color of said saponified solution from a darker color to a lighter color.

5 10. The method of Claim 9 wherein the color of said saponified solution is changed from black to light amber.

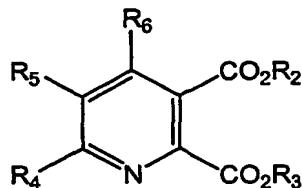
11. The method of Claim 1 wherein said reaction is performed at a temperature of about 60°C to about 110°C.

10 12. The method of Claim 1 wherein said oxidizing agent is added over a time period of about 15 to about 120 minutes.

15 13. The method of Claim 1 wherein said reaction further comprises stirring said saponified solution.

14. The method of Claim 13 wherein said stirring is carried out for a time period of about 15 to about 120 minutes.

20 15. The method of Claim 1 wherein said pyridine-2,3-dicarboxylic acid ester is a compound of the formula



wherein R_4 and R_6 are each independently H, C₁-C₆ alkyl, C₁-C₆ alkenyl, phenyl or substituted phenyl;

25 R_5 is H; halogen; C₁-C₆ alkyl optionally substituted with one or more C₁-C₄ alkoxy groups; C₁-C₆ alkenyl; phenyl or substituted phenyl; and

R_2 and R_3 are each independently C₁-C₆ alkyl, phenyl or substituted phenyl.

16. A method for the in-situ removal of impurities from a solution of pyridine-2,3-dicarboxylic acid ester, said method comprising the steps of:

providing a solution comprising a pyridine-2,3-dicarboxylic acid ester;

saponifying said solution by adding a base, thereby forming a saponified solution

5 comprising a pyridine-2,3-dicarboxylic acid salt;

reacting said solution with an oxidizing agent in an amount effective to remove impurities, thereby providing a purified saponified solution;

adding an acid to said solution, thereby acidifying said solution and converting said pyridine-2,3-dicarboxylic acid salt into the corresponding pyridine-2,3-dicarboxylic

10 acid; and

collecting a purified solution comprising the pyridine-2,3-dicarboxylic acid.

17. The method of Claim 16 wherein said base is a hydroxide.

15 18. The method of Claim 17 wherein said hydroxide is sodium hydroxide.

19. The method of Claim 16 wherein said oxidizing agent is selected from the group consisting of peroxides, peroxyacids, and hypohalite salts.

20 20. The method of Claim 19 wherein said oxidizing agent is hydrogen peroxide.

21. The method of Claim 19 wherein said oxidizing agent is sodium hypochlorite or sodium hypobromite.

25 22. The method of Claim 20 wherein said amount of hydrogen peroxide effective to remove impurities is an amount in the range of about 0.1 to about 2.0 moles hydrogen peroxide per mole of pyridine-2,3-dicarboxylic acid ester.

23. The method of Claim 20 wherein said amount of hydrogen peroxide effective to remove impurities is an amount in the range of about 0.2 to about 0.8 moles hydrogen peroxide per mole of pyridine-2,3-dicarboxylic acid ester.

5 24. The method of Claim 16 wherein said amount of oxidizing agent effective to remove impurities is an amount necessary to change the color of said saponified solution from a darker color to a lighter color.

10 25. The method of Claim 24 wherein the color of said saponified solution is changed from black to light amber.

26. The method of Claim 16 wherein said reaction is performed at a temperature of about 60°C to about 110°C.

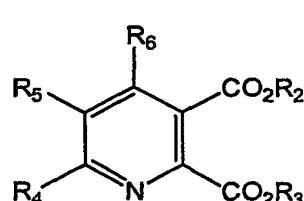
15 27. The method of Claim 16 wherein said oxidizing agent is added over a time period of about 15 to about 120 minutes.

28. The method of Claim 16 wherein said reaction further comprises stirring said saponified solution.

20 29. The method of Claim 28 wherein said stirring is carried out for a time period of about 15 to about 120 minutes.

30. The method of Claim 16 wherein said acid is sulfuric acid.

25 31. The method of Claim 16 wherein said pyridine-2,3-dicarboxylic acid ester is a compound of the formula



wherein R₄ and R₆ are each independently H, C₁-C₆ alkyl, C₁-C₆ alkenyl, phenyl or substituted phenyl;

R₅ is H; halogen; C₁-C₆ alkyl optionally substituted with one or more C₁-C₄ alkoxy groups; C₁-C₆ alkenyl; phenyl or substituted phenyl; and

5 R₂ and R₃ are each independently C₁-C₆ alkyl, phenyl or substituted phenyl.

32. The method of Claim 16, wherein said pyridine-2,3-dicarboxylic acid is 5-methyl-pyridine-2,3-dicarboxylic acid or 5-ethyl-pyridine-2,3-dicarboxylic acid.

10 33. A method for the preparation of herbicidal 2-(2-imidazolin-2-yl)nicotinic acids, esters, and salts, said method comprising the steps of:

providing a solution comprising a pyridine-2,3-dicarboxylic acid ester;
saponifying said solution by adding a base, thereby forming a saponified solution comprising a pyridine-2,3-dicarboxylic acid salt;

15 reacting said solution with an oxidizing agent in an amount effective to remove impurities, thereby providing a purified saponified solution;

adding an acid to said purified saponified solution, thereby acidifying said solution and converting said pyridine-2,3-dicarboxylic acid salt into the corresponding pyridine-2,3-dicarboxylic acid; and

20 using said pyridine-2,3-dicarboxylic acid as an intermediate in the preparation of herbicidal 2-(2-imidazolin-2-yl)nicotinic acids, esters, and salts.

34. The method of Claim 33 wherein said base is a hydroxide.

25 35. The method of Claim 34 wherein said hydroxide is sodium hydroxide.

36. The method of Claim 33 wherein said oxidizing agent is selected from the group consisting of peroxides, peroxyacids, and hypohalite salts.

30 37. The method of Claim 36 wherein said oxidizing agent is hydrogen peroxide.

38. The method of Claim 36 wherein said oxidizing agent is sodium hypochlorite or sodium hypobromite.

5 39. The method of Claim 37 wherein said amount of hydrogen peroxide effective to remove impurities is an amount in the range of about 0.1 to about 2.0 moles hydrogen peroxide per mole of pyridine-2,3-dicarboxylic acid ester.

10 40. The method of Claim 37 wherein said amount of hydrogen peroxide effective to remove impurities is an amount in the range of about 0.2 to about 0.8 moles hydrogen peroxide per mole of pyridine-2,3-dicarboxylic acid ester.

15 41. The method of Claim 33 wherein said amount of oxidizing agent effective to remove impurities is an amount necessary to change the color of said saponified solution from a darker color to a lighter color.

42. The method of Claim 41 wherein the color of said saponified solution is changed from black to light amber.

20 43. The method of Claim 33 wherein said reaction is performed at a temperature of about 60°C to about 110°C.

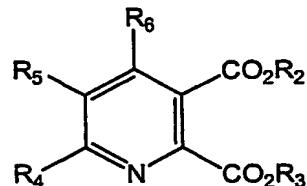
44. The method of Claim 33 wherein said oxidizing agent is added over a time period of about 15 to about 120 minutes.

25 45. The method of Claim 33 wherein said reaction further comprises stirring said saponified solution.

30 46. The method of Claim 45 wherein said stirring is carried out for a time period of about 15 to about 120 minutes.

47. The method of Claim 33 wherein said acid is sulfuric acid.

48. The method of Claim 33 wherein said pyridine-2,3-dicarboxylic acid ester is a compound of the formula



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wherein R₄ and R₆ are each independently H, C₁-C₆ alkyl, C₁-C₆ alkenyl, phenyl or substituted phenyl;

R₅ is H; halogen; C₁-C₆ alkyl optionally substituted with one or more C₁-C₄ alkoxy groups; C₁-C₆ alkenyl; phenyl or substituted phenyl; and

10 R₂ and R₃ are each independently C₁-C₆ alkyl, phenyl or substituted phenyl.

49. The method of Claim 33 wherein said pyridine-2,3-dicarboxylic acid is 5-methyl-pyridine-2,3-dicarboxylic acid or 5-ethyl-pyridine-2,3-dicarboxylic acid.